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A study of the separation of lactose from whey ultrafiltration permeate using nanofiltration

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Abstract

Whey is the main by-product obtained from cheese production. It contains a high concentration of organic matter, mainly proteins and lactose, and mineral salts. Usually, pre-treated whey was discharged into sewer together with the other liquid effluents from the dairy industry. However, the increasingly stringent legal standards for wastewaters in contrast with the high COD and BOD of whey have entailed a change in the approach to whey management. The present paper is focused on the study of the concentration and diafiltration processes applying nanofiltration (NF) membranes for whey after its ultrafiltration. The NF membrane used in all experiments was DS-5 DL from GE-OSMONICS. For each test, different transmembrane pressures ranging between 0.5 and 2.5 MPa were tested.

Results indicated that both the lactose concentration and the whey demineralization were achieved for a combination of the concentration and continuous diafiltration modes. The best operating conditions for the process (those entailing the lowest lactose loss) were 2 MPa and a VDF of around 2. Higher volume dilution factor (VDF) implied higher chloride removal from the whey but at the same time a lactose loss increase.

Keywords: Whey; Lactose; Ultrafiltration; Nanofiltration

1. Introduction

Whey is the main by-product obtained from cheese production. It contains a high concentration of organic matter, mainly proteins and lactose, and mineral salts. A typical whey composition is 5% lactose, 1% proteins and 0.5% ashes [1,2]. Usually, pre-treated whey was discharged into sewer together with the other liquids effluents of the dairy industry. However, the increasingly stringent

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legal standards for wastewaters in contrast with the high COD and BOD of whey have entailed a change in the approach to whey management. Thus, different possibilities of whey valorization were studied and it can be stated that demineralized whey is a good candidate for its application in a great variety of products in food industry such as beverage powders, nutrition bars, soups, bakery, confectionery coatings and desserts (ice cream, frozen dairy).

Conventional whey demineralization is performed in two stages: it is firstly evaporated forming whey concentration and then the concentrated stream is demineralized by electrodialysis and/or ion-exchange. However, these processes need high capital and operating costs [3–5].

Membrane technologies are separation methods used in agro-industries to concentrate and/or purify different streams. Particularly, the dairy industry has been one of the greatest application fields of membrane systems. In this way, in the 1980s, researchers studied the application of membranes for milk concentration in the production of non-standardized cheese. Besides, membrane separation may be particularly attractive to fluid milk processors in the future because it demands little energy and does not destroy any product during treatment. In particular, four basic types of membrane filtrations present potential applications for the dairy industry, i.e. reverse osmosis, nanofiltration (NF), ultrafiltration and microfiltration [6–8].

NF process can separate low molecular weight solutes from mineral salt solutions. Although the separation mechanisms are not clear enough, in general it is determined by complex steric and electrical effects or electroneutrality principle (Donnan Effect) [9]. In addition, its behavior is influenced by both the feed solution and the membrane characteristics [10].

The present paper is focused on the study of the concentration and diafiltration processes applying NF membranes. Separation performance in NF membranes is strongly dependent on the operation mode (for example, concentration and diafiltration) and on the properties of the solutes and of the membrane. Diafiltration is a technique used for the achievement of high purification rates of macro-microsolutes with an economically acceptable permeation flux. The overall process of diafiltration may involve three steps: pre-concentration, diafiltration and post-concentration [11]. Previous works have reported that the performance of the diafiltration step is significantly higher for hydrophilic membranes [12].

Several authors have reported different diafiltration applications. Thus, this operation mode has been applied in the pharmaceutical industry for products purification using NF membranes [13]. Yazdansshenas et al. [14] studied the diafiltration step to treat apple juice using ultrafiltration membranes. Authors used the gel polarization model and optimized the process according to the operating time instead of the retentate concentration.

Referring to the NF application in the dairy industry in concentration mode (CM), it is worth mentioning the whey concentration and demineralization studies carried out by Räsänen et al. [15]. They observed that the concentration polarization layer on the membrane surface grows and the osmotic pressures increase with increasing whey concentration. Lactose loss in the process was of 1%. These authors also performed experiments with Desal 5 membrane obtaining high monovalent chloride ions passage. Suarez et al. [16] studied the concentration step with dairy effluents. They obtained a degree of mineral salt removal by NF of 27–36%, depending on the value of the volume reduction factor (VRF). The NF membrane feeding streams were ultrafiltrated whey and milk.

Minhalma et al. [17] studied the NF process in order to obtain a rich lactose fraction in the concentrate stream and a process water with a high salt content in the permeate stream from cheese whey. Two membranes (NFT50 and HR95 PP) were tested working at two operation modes: total recirculation and concentration. The best results were obtained with NFT50 membrane at 3 MPa allowing a water recovery of 80%.

In the same way, Barrantes and Morr [18] studied the deacidification and demineralization of the cottage cheese whey. They obtained an acid lactic rejection value of 31% and 67.2% for concentration and diafiltration modes, respectively. Regarding mineral salts, rejections for the concentration and the diafiltration modes were of 30% and 71%, respectively.

Although the aforementioned references have studied the whey demineralization, this work goes deeper in the comparison among three different NF operating modes (recirculation, concentration and continuous diafiltration) from the point of view of the lactose and ions rejections and permeates flux.

2. Material and methods

2.1. Whey composition

Ions were analyzed by the ion Chromatograph 790 Personal IC from METROHM (Switzerland) (with columns Metrosep A supp 5 for anions and Metrosep C 2 for anions) according to the Standard Methods [19]. The residual standard deviations ranged between 0.1 and 1% depending on measured ion. COD was analyzed with cell tests (reference 1.14555.0001) from MERCK, Germany. Conductivity and pH were determined by means of the conductimeter GLP32 and the pH meter GLP 22 from CRISON instruments (Spain), respectively. Lactose concentration was determined according to the Spanish standard UNE 34-826-83 [20]. Fats were measured by Gerber Method according to Spanish standard UNE 34-898-86 [21] and proteins were calculated by multiplying the total nitrogen (Spanish standard UNE 34-823-83 [22]) by 6.38.

Table 1
Whey composition of the ultrafiltrated sweet cheese (UF-whey)

Parameters	Value
pH	6.50
Conductivity, mS cm^{-1}	6.40
Proteins, g L^{-1}	2.02
Lactose, g L^{-1}	56.10
Fats, g L^{-1}	0.00
Chlorides, mg L^{-1}	1640
Sodium, mg L^{-1}	460
Potassium, mg L^{-1}	1700
Magnesium, mg L^{-1}	100
Sulphates, mg L^{-1}	110
Calcium, mg L^{-1}	289
Phosphates, mg L^{-1}	550

Fresh whey composition (average concentration values) after its ultrafiltration is shown in Table 1. The aim of the whey ultrafiltration is a protein concentrate recovery. Besides, ultrafiltration prior to NF avoids proteins adsorption onto the NF membrane pores, which could produce membrane fouling.

2.2. NF experiments

Membrane experiments were performed in a laboratory plant equipped with a pressure vessel that contains one spiral wounded membrane element with an active surface of 2.51 m^2. The operating temperature was 16–18°C. Different transmembrane pressures ranging between 0.5 and 2.5 MPa were tested. The NF membrane used in all the experiments was DS-5 DL from GE-OSMONICS. This membrane is made of a thin-film composite (TFC) with polyamide surface on polysulfone support and polyester matrix. Its cut-off ranges between 150 and 300 Da; 98% of lactose retention was measured at 22°C for a feed solution containing 50 g L^{-1} of lactose concentration.

NF experiments were divided into three groups, according to the operation mode (recirculation, concentration and continuous diafiltration). Membrane cleaning consisting in a water rinse was carried out after UF-whey NF. Afterwards, membrane water permeability was checked to ensure the initial flux recovery.

Permeate and solute fluxes, J_P ($L \cdot m^{-2} h^{-1}$) and J_s ($g \cdot m^{-2} h^{-1}$), respectively, and lactose and ion concentrations were measured at steady state conditions. Solute retentions, R_i (%), were calculated according to Eq. (1), where $C_{i,P}$ and $C_{i,b}$ are the solute concentrations in the permeate and feed streams, respectively.

$$R = 1 - \frac{C_{i,P}}{C_{i,b}} \quad (1)$$

2.2.1. Total recycle mode

In these experiments both permeate and concentrate streams were returned to the feed tank so that feed concentration could remain constant.

2.2.2. Concentration mode

The CM was carried out by storing the permeate stream in a separate tank until the feed solution volume was reduced to 50%. The operating pressures were 1.0 and 2.0 MPa. The VRF was determined as the relationship between the initial ($V_{initial}$) and the final (V_{final}) volumes in the feed tank, as shown in Eq. (2).

$$\mathrm{VRF} = \frac{V_{initial}}{V_{final}} \quad (2)$$

The yield values for each individual whey component were calculated as a fraction of its initial concentration (Eq. (3))

$$Y = \frac{C_f}{C_i} \quad (3)$$

where C_f and C_i are the component concentrations at the end and at the beginning of the experiment, respectively.

2.2.3. Continuous diafiltration mode

The continuous diafiltration mode (CDM) was carried out with water addition (at the pH and temperature of the UF-whey) at the same flow rate as the permeate flow rate. Thus, the feed volume was constant during the process. The experiments ended when the feed solution conductivity was reduced to 1000 $\mu S\ cm^{-1}$. The volume dilution factor (VDF) was determined as the relationship between the withdrawn permeate volume ($V_{P,t}$) and the initial feed volume ($V_{feed,i}$) as shown in Eq. (4).

$$\mathrm{VDF} = \frac{V_{P,t}}{V_{feed,i}} \quad (4)$$

This operation mode is used when it is needed to increase the removal efficiency of a component that is only partially retained by the membrane (ions in this case).

Solute removal efficiencies can be calculated considering the concentration values in the diafiltration mode (Eq. (5)).

$$\mathrm{Removal}\% = \frac{C_i - C_f}{C_i} \times 100 \quad (5)$$

where C_i and C_f are the solute concentrations in the initial and the final feed volume, respectively, for a particular VDF.

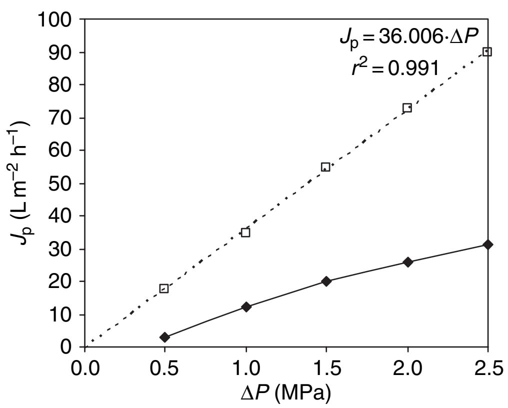

Fig. 1. Permeate flux variation with transmembrane pressure. □, Pure water; ◆, UF-whey.

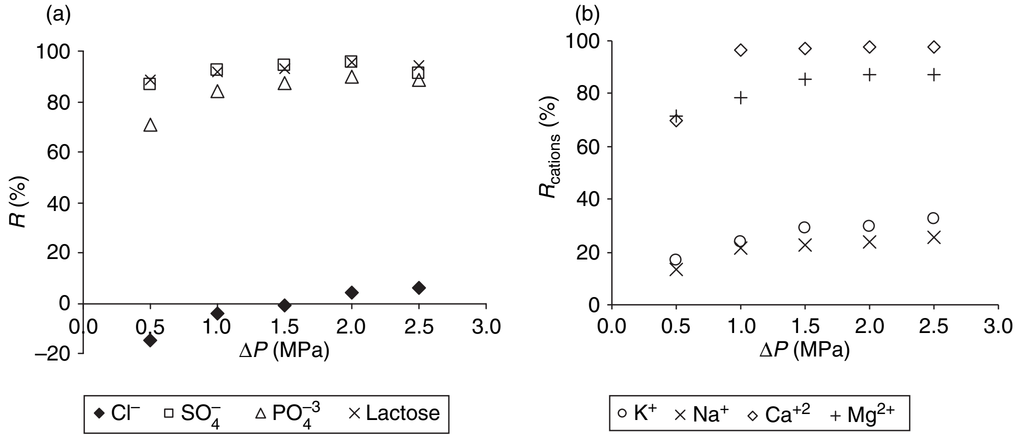

Fig. 2. Solute retention variation with transmembrane pressure.

3. Results and discussion

3.1. Total recycle mode

Previous to NF experiments with UF-whey, membrane permeability was measured with deionized water (conductivity $<5\ \mu S\ cm^{-1}$). The permeability coefficient was $36\ L\ m^{-2} h^{-1} MPa^{-1}$.

3.1.1. Permeate flux

In the experiments with constant concentration of the feeding whey, a linear increase of the permeate flux with the transmembrane pressure was observed as expected. At transmembrane pressures higher than 2.5 MPa, the flux tended to reach a practically constant value as shown in Fig. 1. This fact can be explained as a consequence of the concentration polarization layer formation on the membrane surface or it can be due to the additional membrane resistance caused by the proteins that were not previously removed by ultrafiltration.

Irreversible fouling was not observed in the experiments, since the membrane original flux was recovered after rinsing.

3.1.2. Solute retention

Solutes retention values at different transmembrane pressures are shown in Fig. 2. It can be observed that for transmembrane pressures lower than 1.5 MPa, lower ion rejections were measured. However, ion rejections remained almost constant for higher transmembrane pressures.

The high diffusive transport of salts through the membrane compared to the convective transport explains the low rejection values at low volumetric fluxes. On the contrary, at high volumetric fluxes the convective transport becomes more important and the rejection increases [5,23].

It is worth highlighting the low monovalent ions retention and especially the negative

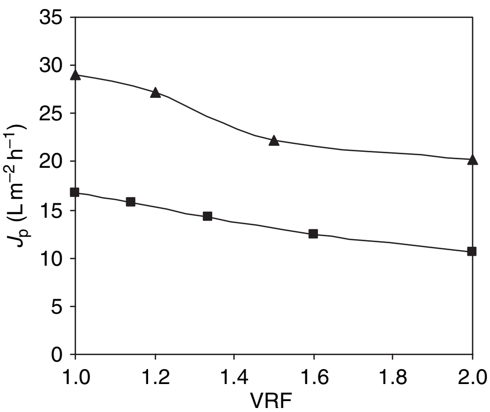

Fig. 3. Influence of the VRF and the transmembrane pressure on permeate flux. ▲, $\Delta P = 2$ MPa; ■, $\Delta P = 1$ MPa.

Table 2
Solute retention values feeds as a function of pressure applied ($\Delta P = 1$ and 2 MPa; VRF = 2)

Feed	Concentration ($mg\,L^{-1}$)				R (%)	
	$\Delta P = 1$ MPa		$\Delta P = 2$ MPa		$\Delta P = 1$ MPa	$\Delta P = 2$ MPa
	Initial	Final	Initial	Final		
Lactose	36,600	57,700	39,100	66,700	84.58	89.06
Ca^{+2}	223.11	334.79	207.55	348.05	72.98	78.44
SO_4^-	99.00	144.47	85.76	149.45	77.37	73.24
PO_4^{-3}	350.50	538.00	522.57	804.30	77.78	81.00
Mg^{2+}	66.02	95.21	48.39	76.89	73.74	80.60
Cl^-	1010.00	868.20	1162.50	1087.35	−18.29	−10.60
Na^+	273.87	323.24	292.44	351.48	26.56	29.13
K^+	1126.90	1260.54	1273.20	1504.80	30.08	33.53

chloride ion rejection [24,25]. These negative retentions (ion concentration in permeate higher than in the feeding stream) were due to the Donnan effect caused by the membrane charge. Thus, chloride ions (the smallest anions in the UF-whey) passed through the membrane in order to maintain the electroneutrality.

3.2. Concentration mode

3.2.1. Permeate flux

In the CM, a gradual permeate flux decrease was observed for increasing feed concentration at a constant transmembrane pressure.

Fig. 3 illustrates the permeate variation flux with the VRF at 1 and 2 MPa. Flux decrease is sharper for 1 MPa (from $17\,L{\cdot}m^{-2}{\cdot}h^{-1}$ to $11\,L{\cdot}m^{-2}{\cdot}h^{-1}$ at 1 MPa and from $29\,L{\cdot}m^{-2}{\cdot}h^{-1}$ to $20\,L{\cdot}m^{-2}{\cdot}h^{-1}$ at 2 MPa). This behavior is explained both by the increase in the osmotic pressure of the feed solution and by concentration polarization phenomena.

3.2.2. Solute retention

In Table 2, characteristics of the feed solution (VRF = 1), the concentrated UF-whey (VRF = 2) and the calculated solute retention indexes can be observed. Among all the studied solutes, it is worth highlighting that the highest separation yield (negative rejection) was for the chloride ions. In the same way, lactose was the solute with the highest retention.

If the ions rejections are compared, polyvalent ones are considerably higher than those obtained for monovalent ions, as expected. The observed rejections went down on increasing the VRF (this is explained by concentration polarization phenomena on the membrane surface).

On the other hand, ions retentions (except for sulphate ions) were higher for 2 MPa. In this way, calcium rejection was 73.0% for 1 MPa and

Table 3
Feeding and permeate streams COD values in CM tests

VRF	COD ($g\,O_2\,L^{-1}$)			
	Feed stream		Permeate stream	
	$\Delta P = 1$ MPa	$\Delta P = 2$ MPa	$\Delta P = 1$ MPa	$\Delta P = 2$ MPa
1.0	47.50	53.25	2.81	1.00
2.0	74.50	78.25	6.00	3.97

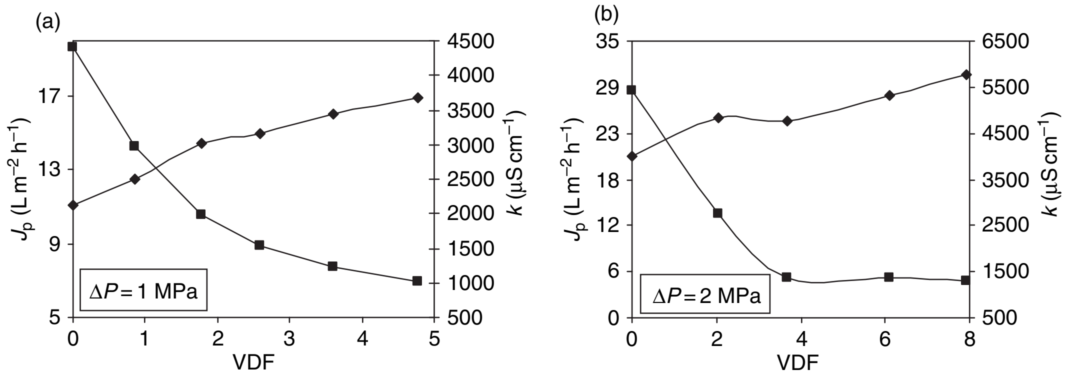

Fig. 4. Evolution of the feed conductivity and the permeate flux in CDM for $\Delta P = 1$ and 2 MPa. ■, Feed conductivity; ◆, J^p ($L\,m^{-2}\,h^{-1}$).

78.4% for 2 MPa. Besides, in order to accomplish the electroneutrality principle, the monovalent ions permeation increased for VRF = 2 at lower transmembrane pressure. Similar results were reported by Rassanen et al. [15] and Suárez et al. [16].

Table 3 summarizes the COD analysis results for the CM experiments for two transmembrane pressure values (1 MPa and 2 MPa). As expected, the COD in the membrane feeding stream increased with the VRF due to the lactose rejection.

3.3. Continuous diafiltration mode

3.3.1. Permeate flux

CDM was used to enhance the salts permeation, although a higher lactose loss could be expected.

During CDM, the permeate flux increased around 55% (from 11 to 17 $L\,m^{-2}\,h^{-1}$) and 27% (from 22 to 30 $L\,m^{-2}\,h^{-1}$) for 1 MPa and 2 MPa, respectively, since the salt concentration in the feed stream decreased and, as a consequence, concentration polarization phenomena

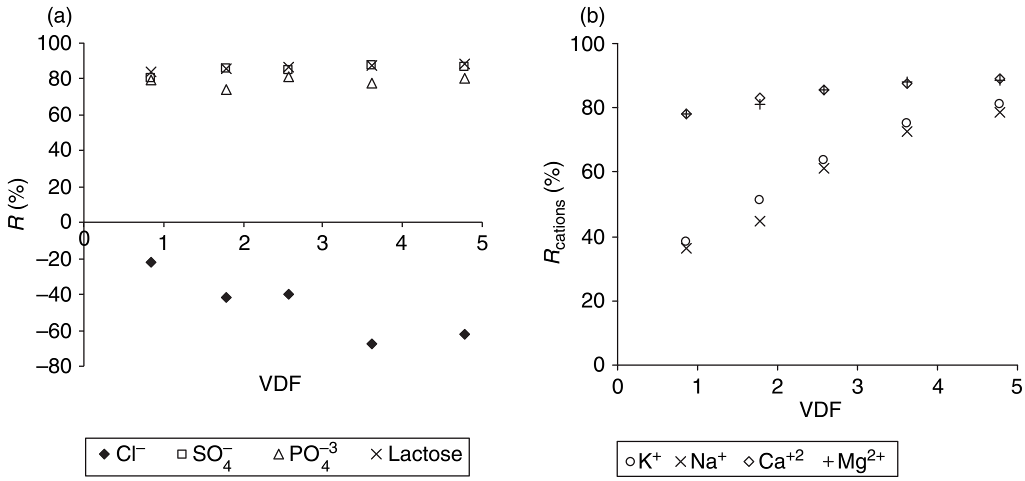

Fig. 5. Solute retention variation with VDF for CDM ($\Delta P = 1$ MPa).

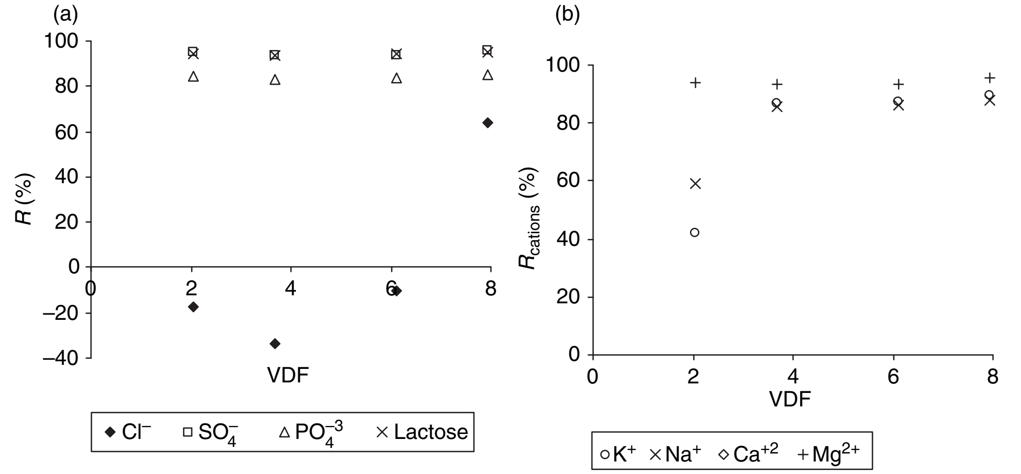

Fig. 6. Solute retention variation with VDF for CDM ($\Delta P = 2$ MPa).

and osmotic pressure effects went down. Thus, conductivity in the feed tank diminished, reaching a minimum value of 1000 and 1500 μS cm^{-1} for 1 MPa and 2 MPa, respectively. This behavior can be observed in Fig. 4.

3.2.2. Solute retention

The lactose retention remained almost constant in the experiments. On the contrary, the monovalent ions retention changed with increasing *VDF* for a particular transmembrane pressure. It can be highlighted that chloride retention was negative due to the Donnan effect as explained above. These results can be observed in Figs. 5 and 6.

According to the results it can be stated that ions retentions increased with the VDF. This increase was sharper for monovalent cations (potassium and sodium) from VDF 2 to 4.

In Figs. 7 and 8 ions concentrations in the feed tank and ions removal efficiencies related to

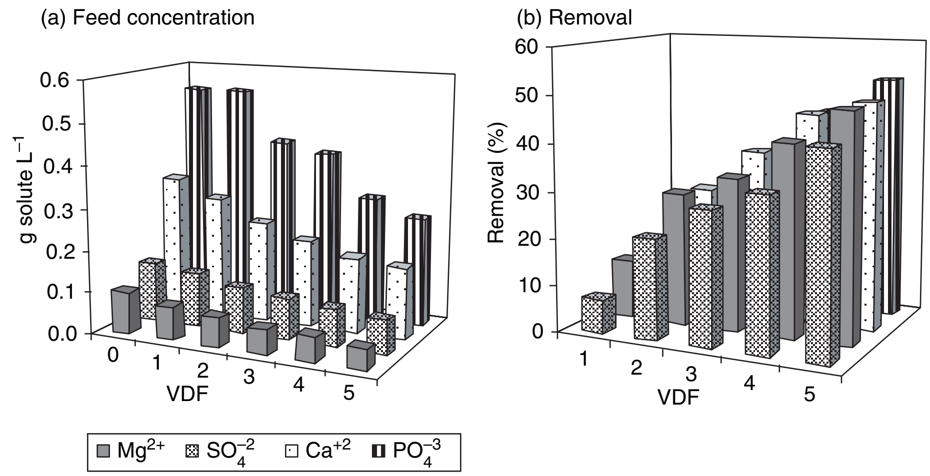

Fig. 7. Polyvalent ions concentration and removal referred to the feed for different VDF ($\Delta P = 1$ MPa).

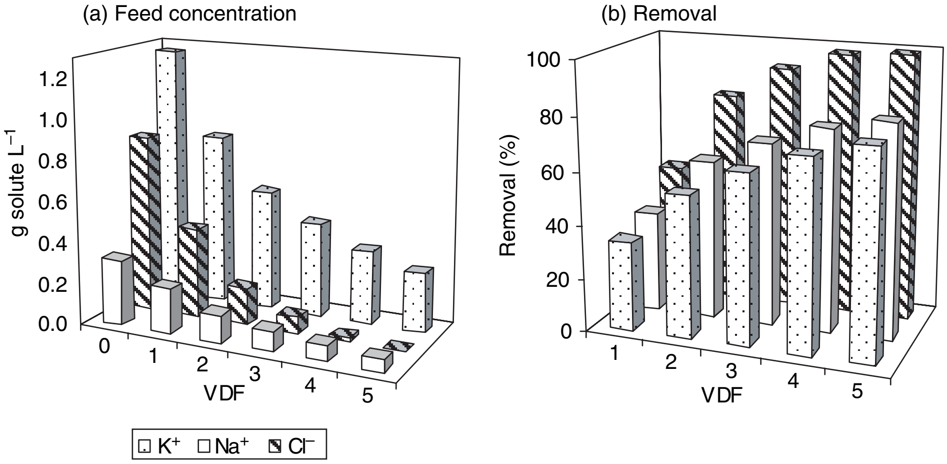

Fig. 8. Monovalent ions concentration and removal referred to the feed for different VDF ($\Delta P = 1$ MPa).

the original UF-whey for the different VDF at 1 MPa are shown.

At 1 MPa, chloride ions concentration was reduced by 99% for a VDF value of 4.78. That means that practically a total chloride ions removal in the feed is achieved. Concerning the sodium and potassium ions, a removal of 80% was reached. However, the polyvalent ions removal was only of approximately 50%.

Figs. 9 and 10 show the ions concentrations in the feed tank and their removal efficiencies related to the original UF-whey for the different VDF at 2 MPa.

Results at 2 MPa indicate a similar tendency as explained for 1 MPa. At 2 MPa the continuous diafiltration process is very efficient to reduce the salt content in the feed stream, with high demineralization ratios.

In Fig. 11, the analytical results for lactose can be observed. At 1 MPa, lactose losses increased with the VDF reaching a value of 36.6% for a VDF of 4.78. Therefore, taking into

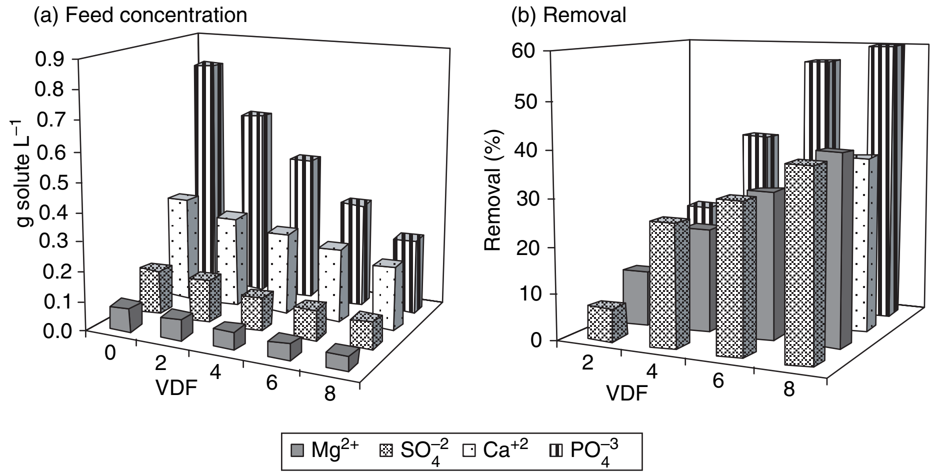

Fig. 9. Polyvalent ions concentration and removal referred to the feed for different VDF ($\Delta P = 2$ MPa).

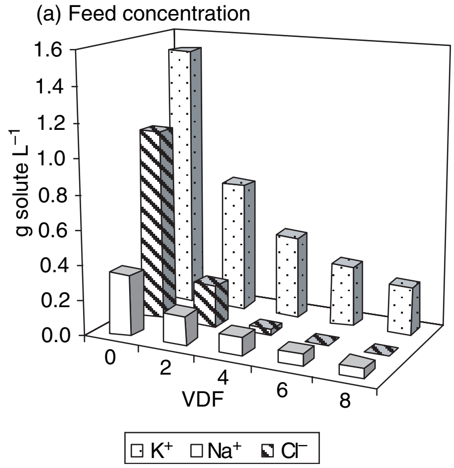

Fig. 10. Monovalent ions concentration and removal referred to the feed for different VDF ($\Delta P = 2$ MPa).

account the results obtained a VDF value of 2.58 as the optimum one for 1 MPa can be considered, since increasing VDF values imply an increase of the lactose losses.

It can be concluded that the best results for 2 MPa occurred at VDF of 2.06, since lactose losses are not very high and the monovalent ions removals were around of 80%.

Concerning COD, its value decreased with the VDF because of the lactose concentration decrease in the feed stream. Besides, it is worth mentioning that the lowest COD values for a particular VDF were obtained at 2 MPa (Fig. 12).

As it occurred for CM, CDM permeate streams cannot be directly discharged into sewer due to the high organic matter content (COD). For that, a further treatment is needed (for example, reverse osmosis). Nevertheless, they can be used in the cheese processing again with a subsequent

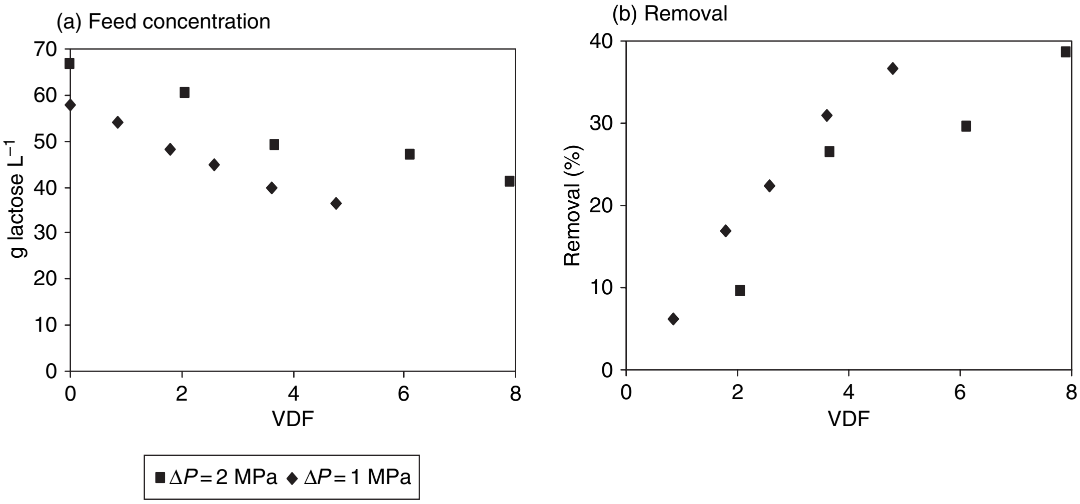

Fig. 11. Lactose concentration and removal referred to the feed for different VDF.

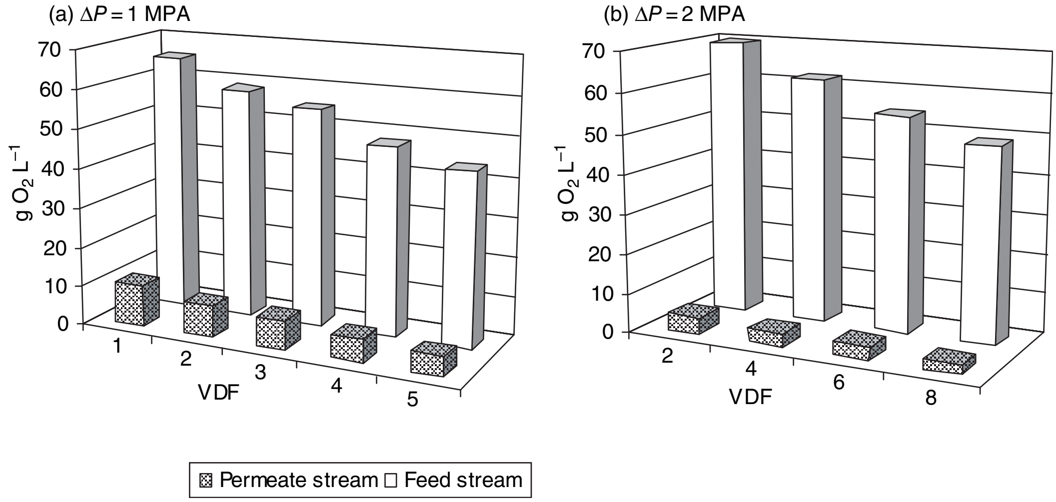

Fig. 12. COD values of feed and permeate streams during CDM operation.

salts and water reuse. Another management possibility could be their use as cleaning waters, like reported by other authors [26,27].

4. Conclusions

NF process was effective for mineral salts removal from whey with the aim of reusing lactose. In this way, three operation modes were studied: total recycle, concentration and continuous diafiltration.

Combining the concentration and CDMs, the lactose concentration and the whey demineralization are achieved. The best operating conditions for the process (those entailing the lowest lactose loss) were 2 MPa and a VDF of around 2. Higher VDF implied higher chloride removal from the whey but at the same time a lactose loss increase.

A further treatment of the NF permeate streams is required in order to discharge it into sewer because of their COD measured values. However, permeates can be reused in cheese processing.

Fouling problems were not detected for the performed NF tests. However, longer experiments in a larger scale plant have to be performed to study the membrane life expectance and to evaluate the economical feasibility of the process.

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